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Process for the preparation of mono- and bis(fluoroalkyl)phosphoranes
and the corresponding acids and phosphates

This application is a 371 of PCT/EP04/11940 filed on 10/22/2004.

5 The present invention relates to a process for the preparation of mono-
(fluoroalkyl)- or bis(fluoroalkyl)phosphoric acids, mono(fluoroalkyl) or bis-
(fluoroalkyl) phosphates and the corresponding phosphoranes thereof.

10 A process known from the prior art for the synthesis of fluoroalkylphos-
phoranes is based on the electrochemical Simons fluorination (ECF) of
alkylphosphines (N. Ignatyev, P. Satori, *J. of Fluorine Chem.*, 103 (2000)
57-61; WO 00/21969) and, owing to the high yields, is particularly suitable
for the synthesis of tris(fluoroalkyl)difluorophosphoranes. In the electro-
chemical fluorination of dialkylphosphines having short alkyl chains (having
15 less than C₄), the yield of the corresponding perfluorinated phosphoranes
is significantly lower.

20 The tris(fluoroalkyl)difluorophosphoranes can be used as starting materials
for the synthesis of various phosphates (WO 98/15562, DE 196 41 138,
EP 1 127 888) and a novel tris(fluoroalkyl)trifluorophosphoric acid
(DE 101 30 940). This acid can be used not only for the synthesis of vari-
ous salts, but can also be hydrolysed to give the corresponding bis(fluoro-
alkyl)phosphinic acid (DE 102 169 97). Bis(fluoroalkyl)phosphinic and
25 fluoroalkylphosphonic acid and salts thereof can also be obtained by
hydrolysis of tris(fluoroalkyl)difluorophosphoranes (DE 102 169 95).

30 A process known from the prior art for the preparation of mono(perfluoro-
alkyl)- and bis(perfluoroalkyl)fluorophosphoranes is furthermore a multistep
reaction based on the reaction between phosphorus and perfluoroalkyl
halides, which are very expensive (T. Mahmood, J.M. Shreeve, *Inorg.*
Chem., 25 (1986) 3128). This reaction frequently requires high pressures
35 and temperatures.